AD-781 933

OPTICAL AND ELECTRON MICROSCOPE STUDIES OF THE CATALYSIS OF COMBUSTION OF AMMONIUM PERCHLORATE

O. P. Korabeinichev, et al

Army Foreign Science and Technology Center
Charlottesville, Virginia

8 August 1973

AD781933

DEPARTMENT OF THE ARMY

U.S. ARMY FOREIGN SCIENCE AND TECHNOLOGY CENTER
220 SEVENTH STREET NE.
CHARLOTTESVILLE, VIRGINIA 22901

In Reply Refer to:
FSTC-HT-23-1772-73
DIA Task No. T70-23-01

Date: 8 August 73

TRANSLATION

ENGLISH TITLE: Optical ~~Microscopic~~ and Electron Microscope Studies of the Catalysis of Combustion of Ammonium Perchlorate

FOREIGN TITLE: Mikroskopicheskoye i Elektronnomikroskopicheskoye Issledovaniye Kataliza Goreniya Perkhlorata Ammoniya

AUTHOR: O. P. Korabeynichev, I. A. Ryzhak. P. N. Kuznetsov

SOURCE: Fizika Goreniya i Vzryva, Vol 8, 1972, pp 323-4.

LANGUAGE: Russian

TRANSLATOR: Albert L. Peabody, Leo Kanner Associates

REQUESTOR: AMXBR-XA-FI

Earlier works [1,2], have published results of studies produced primarily using the scanning electron microscope on the structure of the extinguished combustion surface of ammonium perchlorate (APC) and fuels based on it. The literature contains no data on the dispersion and structure of the catalysts in APC specimens, or on the combustion surface of extinguished specimens. This information could shed light on the mechanism of the action of catalysts during combustion of APC. The present work presents the results of microscopic and electron microscope studies of the structure and dispersion of the catalysts Fe_2O_3 and CuO in specimens of APC and on the combustion surface of extinguished specimens.

The replica method was used in the study. A thin layer (a few hundred Angstroms) of carbon was sputtered ~~poured~~ onto the specimen surface, and it adhered to the catalyst particles located on the surface. After dissolution of the specimen in water, what remained was a carbon film carrying the catalyst particles, which was then studied under the electron microscope at magnifications of 3000-10000. The structure of the surface was also studied with a type MBI microscope with a magnification of about 100.

4

The burning APC specimens with catalysts were extinguished by the thermal method (by heat transfer at the point of contact of the side surface of the specimen with metal plates). Iron oxide with a surface of 120 m^2/g was produced by heating of the hydroxide. The copper powder had a specific surface of about 1 m^2/g. The catalyst, in a quantity of about 2%, and the APC, 50-100 μ fraction, were carefully mixed and pressed under pressure of 3000 kg/cm^2.

In order to study changes in the structure and activity of the catalyst during the process of combustion, catalyst particles were selected which had been present in the combustion zone (the products of combustion struck a cold, moist surface, on which the solid particles were held).

Figures 1a and b show electron microscope photographs of the cleavage surface of a specimen of APC + 2% Fe_2O_3. As measurements showed, the catalyst particles (measuring from 100 to 0.01 μ) were widely and unevenly dispersed through the volume of the APC. The large particles resulted from sticking together of smaller particles. Figure 1 clearly shows the block structure of the cleavage surface of the APC specimen. The dimensions of the blocks are a few microns. Figure 2 shows electron microscope photographs of the Fe_2O_3 catalyst, both initial (a) and after taking part in combustion and being removed from the combustion zone (b). As we can see from the figure, during the process of combustion the catalyst particles become spherical in shape with a mean diameter of about 0.05 μ, apparently as a result of sintering (since the combustion temperature of APC, about 1000° C, is less than the melting point of Fe_2O_3, about 1500° C). The specific surface of catalysts which have taken part in combustion, defined by the method of thermal desorption of argon, is decreased by a factor of 4 (to 30 m^2/g).

Figure 3 a, b shows electron microscope photographs of the cleavage surface of an initial specimen of PCA + 2% CuO and the extinguished combustion surface of the same specimen at 1 atm. Figure 3 gives us an idea of the dispersion and evenness of distribution of CuO catalyst particles in the volume of the APC specimen. In contrast to the Fe_2O_3, in the case of CuO, the initial specimen shows no clumps of particles and their distribution is evener over the surface of the APC particles. The shape and structure of the catalyst particles on the extinguished surface of the specimen differ sharply from the initial shape and structure. Melting occurs on the combustion surface; melting also occurred under the electron beam if a high intensity beam was used. The small drops agglomerate into larger drops, up to several tens of microns, as we can see from microscope pictures with magnifications of about 100. The CuO catalyst enters a chemical reaction with the products of decomposition of APC forming chloride-copper compounds [3] (as follows from the data of thermodynamic and chemical analysis), which melt and evaporate at 400-500° C.

Thus, the data which we have presented indicate that at least two models of combustion are possible for Fe_2O_3 and CuO catalysts. In the first case, heterogeneous catalysis by solid particles occurs; in the second case, catalysis is by liquid particles and homogeneous in the gas phase. In the second case, in constructing a model of combustion catalysis, we must consider processes of agglomeration of catalyst particles on the combustion surface, while in the first case we must consider the heterogeneity of distribution of catalyst particles in the volume of the APC specimen, arising in the process of preparation of specimens, and the change in specific surface and shape of the catalyst particles during the process of combustion.

BIBLIOGRAPHY

1. T. L. Boggs. AIAA — paper, 1969, № 69–142.
2. L. D. Hightower, E. W. Price. Astronautica Acta, 1968, 14; 11–21.
3. О. П. Коробейничев, А. В. Шкарин, А. С. Шмелев. Третий всесоюзный симпозиум по горению и взрыву. Черноголовка, 1971.

Thus, the data which we have presented indicate that at least two models of combustion are possible for Fe_2O_3 and CuO catalysts. In the first case, heterogeneous catalysis by solid particles occurs; in the second case, catalysis is by liquid particles and homogeneous in the gas phase. In the second case, in constructing a model of combustion catalysis, we must consider processes of agglomeration of catalyst particles on the combustion surface, while in the first case we must consider the heterogeneity of distribution of catalyst particles in the volume of the APC specimen, arising in the process of preparation of specimens, and the change in specific surface and shape of the catalyst particles during the process of combustion.

BIBLIOGRAPHY

1. T. L. Boggs. AIAA — paper, 1969, № 69—142.
2. I. D. Hightower, E. W. Price. Astronautica Acta, 1968, 14; 11—21.
3. О. П. Коробейничев, А. В. Шкарин, А. С. Шмелев. Третий всесоюзный симпозиум по горению и взрыву. Черноголовка, 1971.

3. O. P. Korobeynichev, A. V. Shkarin, A. S. Shmelev, Tretiy vsesoyuznyy simpozium po goreniyu i vzryvu [Third All-Union Symposium on Combustion and Explosion]. Chernogolovka, 1971.